# TWO-STAGE COPROCESSING OF WASTE PLASTICS WITH COAL

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### INTRODUCTION

Commingled post-consumer plastic waste (CP#2) or high density polyethylene (HDPE) was found to be thermally degraded only at temperatures higher than 430°C, while typical coal liquefaction temperatures are about 400°C. This causes difficulty for coprocessing coal with waste plastic in single stage at appropriate reaction conditions. Moreover, it is hard to find a catalyst which is effective for depolymerization of both coal and HDPE or CP#2. In this study, two-stage coprocessing was developed to produce liquid fuels. In the first stage, CP#2 or HDPE was iliquefied in an 150-cm³ autoclave reactor at 435°C, 1 hr, with stirring speed of 800 rpm, under N<sub>2</sub> or H<sub>2</sub> with and without catalyst (HZSM-5). The resulting liquid products (yields of 78-88 wt%) were utilized as solvents for liquefaction of DECS-6 coal or Fe loaded DECS-6 coal in tubing reactors at 400°C, 1 hr, 160 rpm, ~2000 psig H<sub>2</sub>.

The conversion of coal to liquids is generally perceived to proceed via free-radical mechanisms. Reactive radical fragments are formed by thermally rupturing scissile bonds and by hydrogenolysis [1]. Once formed, fragments are either stabilized by hydrogen addition or recombined to form regressive, polymeric products. Two external sources of hydrogen are available to meet these demands. These are donor hydrogen in the solvent and gaseous molecular hydrogen. Therefore, solvent quality plays a significant role in determining which reaction path is taken during coal liquefaction. The solvents investigated were mostly hydroaromatics (tetralin, and their combinations [2-6]. Recently, phenolic compounds (phenol, cresol) were also used as coal liquefaction solvents [7]. These solvents are relatively expensive, therefore we chose Plastic-Derived-Liquids (PDL) as a solvent for coal liquefaction.

For coal liquefaction, iron-based catalysts, because of obvious economic and environmental benefits, have been widely studied recently [8-13]. Yuen [8] investigated the effects of various iron precursors on liquefaction of DECS-6 and DECS-17 coals in absence of solvent. As an iron precursor, ammonium iron (III) sulfate dodecahydrate was found to be very effective in increasing the liquid yields of catalytic hydroliquefaction of Blind Canyon coal. Derbyshire [14] also reported that in the presence of solvent, there are the advantages of adding the precursor by impregnation over its addition in the form of particulates. Thus, a DECS-6 coal, impregnated with Fe catalyst, was selected for liquefaction studies in the second stage. Noncatalytic reactions were also examined for comparison.

# **EXPERIMENTAL**

High-volatile bituminous Blind Canyon DECS-6 coal, obtained from the Penn. State Coal Sample Bank, was ground to pass through a 100 mesh Tyler series screen using a ball mill grinder under nitrogen. Ground coals were dried under vacuum at 100°C for six hours, kept overnight at room temperature, stored in glass bottles sealed with nitrogen, and then put in a refrigerator for future use. Iron loaded DECS-6 coal was prepared by incipient wetness impregnation. Ammonium rion (III) sulfate dodecahydrate (AFS(III)), obtained from Aldrich Chemical Company, was used as precursor of iron. After impregnation, the resulting coal was dried at the same conditions as mentioned above. The weight ratio of iron to moisture-free coal was 1.12:100.

HDPE (M.W.=125,000) in bead form was purchased from Aldrich Chemical Company. Commingled post-consumer plastic (CP#2), obtained from the American Plastics Council, was ground to -25 mesh. Detailed analyses of DECS-6 coal and CP#2 are listed elsewhere [15]. Synthesized according to U.S. Patent 4,250,345 [16], HZSM-5 catalyst contains 35 wt%  $Al_2O_3$  binder. HZSM-5, with a Si/Al mole ratio of 35, was pulverized to -100 mesh and calcined in air at 500°C for 3 hours before use.

Depolymerization of HDPE or CP#2 was carried out in a 150-cm<sup>3</sup> stainless steel autoclave (Autoclave Engineers). A mixture of HDPE or CP#2 (20.0000 g) and catalyst (0.4000 g HZSM-5) were charged into the reactor. For reaction under N<sub>2</sub>, the reactor was purged with N<sub>2</sub> 5 times

and then closed with a zero pressure gauge value. For reactions with  $H_2$ , the reactor was pressurized with 1000 psig  $H_2$  after being purged with  $N_2$  at room temperature. The fixed reaction conditions were: 435°C, 60 minutes, 800 rpm. When the reaction was finished and the reactor cooled to room temperature, the gases were collected. The detailed procedure is described elsewhere [15]. Liquid and solid products were separated by filtration at room temperature. The solid portion was washed with excess pentane and then dried at 60°C under vacuum overnight. The solid yield is defined as (weight of solid)x100/(weight of feed), while oil yield is defined as  $\{100 - \text{gas} \text{ yield} - \text{solid} \text{ yield} \}$ . This oil (Plastic-Derived-Liquids) was utilized as solvent in the next stage. The weight of feed refers to weight of HDPE or CP#2. All experiments were performed in duplicate; repeatability of the results was  $\pm 1.5\%$  for oil yield.

In the 2nd stage, solvent (Plastic-Derived-Liquids , tetralin, or waste oil) and dried or iron loaded DECS-6 coal were fed into the  $27\text{-cm}^3$  tubing reactors with a ratio of 2:1 (solvent : dry coal, by weight). The reaction parameters were  $400^\circ\text{C}$ ,  $\sim\!2000$  psig  $H_2$ , 60 minutes, 160 rpm. The reaction procedure was identical to that described in reference 15. As gaseous products came from both coal and solvent, it was difficult to determine how much gas was from coal depolymerization. Therefore, the reaction products were lumped as oil+gas, asphaltenes+preasphaltenes, and THF insolubles. The total conversion is defined as  $\{100x[1-(\text{weight of THF soluble but pentane insoluble}]\}$  and the asphaltenes+preasphaltenes yield as  $\{(\text{weight of THF soluble but pentane insoluble})(100x[1-(\text{weight of pentane insoluble}))]$ . The oil+gas yield is  $\{100x[1-(\text{weight of pentane insoluble}))$ 

The gases obtained from the first stage were analyzed by a flame ionization detector on gas chromatography (HP-5890II) using a column packed with HayeSep Q. The liquid products were analyzed by GC/MS using a 30-m long DB-5 capillary column. The boiling point distribution of the liquid products from the second stage of two-stage processing were determined by modified simulated distillation according to ASTM D 2887-89 and D5307-92. The analysis was performed on HP-5890 series II gas chromatograph, using a Petrocol B column (6 inches long and 0.125 inches outside diameter).

### RESULTS AND DISCUSSION

Degradation of HDPE and CP#2 to Obtain Plastic-Derived-Liquids. For degradation of HDPE, the maximum oil yield, 87.2%, was obtained under N<sub>2</sub> without catalyst, while the maximum gas yield, 21.2%, was produced over HZSM-5 under hydrogen (Table 1). For non-catalytic decomposition of HDPE, the solid yield obtained by reaction under nitrogen was 5.5%, while the same yield obtained under hydrogen it was 15.1%. Thermal depolymerization of HDPE was favored in N<sub>2</sub>. Since hydrogen is a kinetic chain transfer agent; it might saturate the thermally cracked radicals and prevent them from cracking further. Catalytic reaction over HZSM-5 with hydrogen (Table 1) gave the highest gas yield (21.2%) and lowest solid yield (1.1%). The hydrogenation ability of HZSM-5 was also demonstrated by conversion of ethene [17].

Thermal degradation of CP#2 under  $N_2$  gave the highest oil yield, 86.2%, while catalytic reaction over HZSM-5 under  $H_2$  led to the highest gas yield, 17.6%. The cracking or hydrocracking ability of HZSM-5 is reflected in the resulting lower solid yields and higher gas yields, compared with those from thermal reactions (Table 1). Compared with degradation of CP#2 under nitrogen, the non-catalytic reaction under hydrogen gave higher solid yield and less gas and oil yields (Table 1). This means that hydrogen also inhibited thermal decomposition of CP#2 to some extent.

Upon detailed GC/MS analyses, the oil products were categorized into five groups: 1-olefins, n-paraffins, aromatics, naphthenes, and others (Figure 1), where "others" included mainly iso-paraffins, cyclic-olefins, branched and normal internal olefins. For HDPE, compared with oil products from the thermal reaction under  $N_2$ , the oil obtained from non-catalytic reaction under  $H_2$  contained higher n-paraffins and 1-olefins, with lower naphthenes, aromatics and others. Compared with the thermal runs, catalytic reactions over HZSM-5 under both  $N_2$  and  $H_2$  produced oil products with large amounts of aromatics, naphthenes, and others at the expense of 1-olefins and n-paraffins (Figure 1). This is consistent with a similar observation reported previously [18]. Therefore, shape selective zeolite, HZSM-5, had not only high cracking (or hydrocracking) ability, but also had cyclization and aromatization functions.

For degradation of CP#2 under nitrogen, like HDPE degradation under N<sub>2</sub>, the same change in oil composition was observed when HZSM-5 was added, i.e., more aromatics and naphthenes, less I-olefins and n-paraffins. Obviously, HZSM-5 had a stronger effect on HDPE than on CP#2. This may be due to a negative effect of the heteroatoms and trace metals contained

in CP#2. For degradation of CP#2 under hydrogen, the general trends of non-catalytic and catalytic reactions were like those under nitrogen (Table 1, Figure 1).

Plastic-Derived-Liquids Used as Solvents for Coal Liquefaction. Table 2 shows the effects of different solvents on liquefaction of DECS-6 coal at 400°C, 2000 psig H<sub>2</sub>, for 60 minutes. Compared with the non-solvent reaction, reactions in the presence of A-1, A-2, A-7, and A-8 oil gave higher total conversions and gas+oil yields. However, A-4, A-5, A-10, and A-11 oil, which were derived from degradation of CP#2, had slightly negative effects on total conversion although increased gas+oil yields. In the presence of waste oil, total conversion increased to some extent, with slightly increasing gas+oil yield. The positive effect of waste oil may be due to a higher content of metals, which may behave as catalysts at reaction conditions [19]. Tetralin, an excellent hydrogen donor solvent, gave the highest conversion and gas+oil yield. The difference in conversion corresponding to different solvents is significant, with 81.4% for tetralin, 57.1% for waste oil, 39.9-48.4% for Plastic-Derived-Liquids, and 44.0% for the non-solvent reaction.

In Table 3, the catalytic results, using PDL as solvents, are compared with those when tetralin and waste oil were used, and when no solvent was used. Compared with thermal reactions (Table 2), the trend of the effects of solvents on conversion and yields for catalytic runs is the same. A-1, A-2, A-7, and A-8 oil were active for catalytic coal liquefaction, increasing both total conversion and yields, while A-4, A-5, and A-11 oil (obtained from CP#2), decreased total conversion slightly, although enhanced the gas+oil yields greatly. Compared with thermal reactions, the difference in conversion corresponding to different solvents decreased in the presence of catalyst. From Tables 1 and 2, at a catalyst loading of 1.12%, the difference in coal conversions between tetralin and PDL is only about 2-10 percentage points, while the difference in thermal conversions for these solvents is about 33-41 percentage points. This implies that PDL can be used with this Fe catalyst, and results obtained are nearly the same as those obtained from tetralin, an expensive hydrogen donor for coal liquefaction. Compared with the thermal reactions, the total conversions and yields were higher in the presence of iron catalyst for each solvent used. This shows that the Fe catalyst was active for the reaction system at conditions used.

For thermal reactions (Table 4), compared with oil from the non-solvent run, the oil products obtained from the reactions with PDL as solvent contained higher amounts of lower-boiling fractions (gasoline, kerosene, and gas oil), except for A-7 oil, which was produced from degradation of HDPE under hydrogen. It is notable that A-7 oil gave highest conversion and oil+gas yield among PDL (Table 2). The lightest oil was from reaction with A-2 oil as solvent, with 92.1% lower-boiling fractions (b.p. up to 325°C). The quality of oil products obtained from reaction with waste oil as solvent was very poor, although the total conversion was increased to some extent. The lower-boiling fractions (b.p. up to 325°C) was only 14.0%. This indicated that further severe upgrading of the oil products would be required.

For catalytic reactions (Table 5), addition of each PDL improved the quality of oil products, producing more lighter components in oil fractions. The oil product with best quality was obtained with A-8 oil, produced from decomposition of HDPE using HZSM-5 as catalyst under hydrogen. This oil contains 59.6% gasoline fraction (<200°C), 24.3% kerosene fraction (200-275°C), and 7.9% gas oil fraction (275-325°C).

Compared with the corresponding thermal reactions, catalytic reactions gave heavier oil products except for the reaction using A-8 oil as solvent. For example, for the non-solvent reaction, the oil products contain 67.5% and 48.1% lower-boiling fractions (b.p. up to 325°C) for thermal and catalytic reactions, respectively. The same numbers for the reaction with A-1 oil as solvent were 78.4% and 70.3% for thermal and catalytic reactions, respectively. This indicated that the total conversion and gas+oil yield increased for catalytic reactions at the expense of oil quality, although the quality just slightly decreased and conversion greatly improved.

Taking into consideration conversion, oil quality, process economics, and process safety, a reasonable senario is: in the first stage, HDPE or CP#2 is degraded under nitrogen; in the second stage, oil products obtained from the first stage (A-I oil from HDPE and A-4 oil from CP#2) are used as solvents for liquefaction of iron loaded DECS-6 coal. With A-1 oil as solvent, the total conversion of coal was 85.0%, and oil products contained 26.9% gasoline (<200°C), 27.5% kerosene (200-275°C), 15.9% gas oil (275-325°C), 16.6% gas heavy gas oil (325-400°C), and 13.1% vacuum gas oil (400-538°C). The coal conversion was 77.9% in the presence of A-4 oil, and oil products contained 72.0% lower-boiling fractions (b.p. up to 325°C).

## CONCLUSIONS

- HDPE and CP#2 can be thermally or catalytically depolymerized under either nitrogen or hydrogen at 435°C. Thermal reactions gave better results with nitrogen than with hydrogen, while catalytic reactions (HZSM-5 used as catalyst) produced oil products with higher quality under hydrogen than under nitrogen.
- The oil from degradation of HDPE or CP#2 can be used as coal liquefaction solvents. In the
  presence of the impregnated iron catalyst, the Plastic-Derived-Liquids (oil obtained from
  decomposition of HDPE at 435°C, 0 psig initial nitrogen pressure, 1 hour) produced similar
  gas+oil yield and total conversion as did tetralin. The resulting oil contained 70.3% lower
  boiling fractions (b.p. up to 325°C).
- The bench-scale experiments showed that two-stage coprocessing is a feasible and promising
  method for utilization of plastic waste. In the first stage, plastics can be degraded alone under
  nitrogen at 435°C; the resulting liquids can be utilized as solvent for liquefaction of Fe loaded
  coal at 400°C in the second stage.

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Table 1. Yields of Products Obtained from Thermal and Catalytic Degradation of HDPE or CP#2 in a 150-cm<sup>3</sup> Autoclave at 435°C, 60 Minutes, 800 rpm (0.4 g HZSM-5 was used as catalyst for 20.0 g HDPE or CP#2)

Initial Pressure	Run Number	Reaction System	Gas Yield, wt%	Oil Yield, wt%	Solid Yield, wt%
0 psig N <sub>2</sub>					
o poig 112	A-1	HDPE	7.3	87.2	5.5
	A-2	HDPE+2% HZSM-5	16.5	82.1	1.4
	A-4	CP#2	9.3	86.2	4.5
	A-5	CP#2+2% HZSM-5	12.5	83.4	4.1
1000 psig H <sub>2</sub>					
	A-7	HDPE	3.1	81.8	15.1
	A-8	HDPE+2% HZSM-5	21.2	77.8	1.1
	A-10	CP#2	7.5	83.7	8.8
	A-11	CP#2+2% HZSM-5	17.6	76.2	6.2

Table 2. Yields (refer to coal alone) of Products Obtained from Liquefaction of DECS-6 Coal with Plastic-Derived-Liquids as Solvents in a 27-cm<sup>3</sup> Tubing Reactor at 400°C, ~2000 psig H<sub>2</sub>, 60 Minutes, 160 rpm (solvent: dry coal = 1:1, weight)

Solvent	Gas+Oil Yield, wt%	Asphaltenes+ Preasphaltenes Yield, wt%	Conversion, wt%
None	24.4	19.5	43.9
Tetralin	40.2	41.2	81.4
Waste Oil	26.6	30.5	57.1
A-1 Oil (1st stage: HDPE, N <sub>2</sub> )	28.9	18.6	47.5
A-2 Oil (1st stage: HDPE+2% HZSM-5, N <sub>2</sub> )	31.2	17.2	48.4
A-4 Oil (1st stage: CP#2, N2)	30.5	9.4	39.9
A-5 Oil (1st stage: CP#2+2% HZSM-5, N <sub>2</sub> )	27.5	13.9	41.4
A-7 Oil (1st stage: HDPE, H <sub>2</sub> )	34.1	14.3	48.4
A-8 Oil (1st stage: HDPE+2% HZSM-5, H <sub>2</sub> )	28.5	18.6	47.1
A-10 Oil (1st stage: CP#2, H2)	28.4	13.2	41.6
A-11 Oil (1st stage: CP#2+2% HZSM-5, H <sub>2</sub> )	25.8	17.0	42.8

Table 3. Yields (refer to coal alone) of Products Obtained from Liquefaction of Fe Loaded DECS-6 Coal (Fe:dry coal =1.12:100, in weight) with Plastic-Derived-Liquids as Solvents in a 27-cm<sup>3</sup> Tubing Reactor at  $400^{\circ}$ C, ~2000 psig H<sub>2</sub>, 60 Min., 160 rpm (solvent : dry coal = 1:1, weight)

Solvent	Gas+Oil Yield, wt%	Asphaltenes+ Preasphaltenes Yield, wt%	Conversion, wt%
None	29.6	49.6	79.2
Tetralin	41.8	45.1	86.9
Waste Oil	37.9	42.1	80.0
A-1 Oil (1st stage: HDPE, N2)	39.1	45.9	85.0
A-2 Oil (1st stage: HDPE+2% HZSM-5, N <sub>2</sub> )	36.1	48.6	84.7
A-4 Oil (1st stage: CP#2, N2)	42.5	35.4	77.9
A-5 Oil (1st stage: CP#2+2% HZSM-5, N <sub>2</sub> )	36.5	41.1	77.6
A-7 Oil (1st stage: HDPE, H <sub>2</sub> )	39.8	42.7	82.5
A-8 Oil (1st stage: HDPE+2% HZSM-5, H <sub>2</sub> )	42.3	38.4	80.7
A-10 Oil (1st stage: CP#2, H2)	37.9	42.0	79.9
A-11 Oil (1st stage: CP#2+2% HZSM-5, H <sub>2</sub> )	35.2	41.1	76.3

Table 4. Boiling Point Distribution (numbers in weight percent) of Final Oil Products Obtained from Two-Stage Coliquefaction of DECS-6 Coal with HDPE or CP#2 (in the first stage, HDPE or CP#2 was degraded in a 150-cm³ autoclave at 435°C, 1 hour, 800 rpm, under N<sub>2</sub> or H<sub>2</sub>; in the second stage, DECS-6 coal was liquefied with Plastic-Derived-Liquids obtained from the 1st stage as solvents in 27-cm³ tubing reactors at 400°C, ~2000 psig H<sub>2</sub>, 60 minutes, 160 rpm (solvent: dry coal = 2:1, weight ratio))

Solvent	Gasoline (<200°C)	Kerosene (200-275°C)	Gas Oil (275-325°C)	Heavy Gas Oil (325-400°C)	Vacuum Gas Oil (400-538°C)	Vacuum Residue (>538°C)
None	12.6	33.2	21.7	17.0	15.4	0.1
Waste Oil	2.9	5.0	6.1	23.0	47.2	15.8
A-1 Oil	29.6	31.5	17.3	14.6	7.0	0.0
A-2 Oil	49.4	31.4	11.3	6.0	1.9	0.0
A-4 Oil	36.9	31.4	15.5	11.6	4.6	0.0
A-5 Oil	39.4	25.2	12.7	13.5	9.2	0.0
A-7 Oil	24.0	22.6	16.0	16.9	17.3	3.2
A-8 Oil	54.1	23.7	9.1	8.4	4.7	0.0
A-10 Oil	32.7	30.1	16.7	14.0	6.5	0.0
A-11 Oil	46.6	31.2	12.0	7.9	2.3	0.0

Table 5. Boiling Point Distribution (numbers in weight percent) of Final Oil Products Obtained from Two-Stage Coliquefaction of Fe Loaded DECS-6 Coal (Fe: dry coal = 1.12:100, weight ratio) with HDPE or CP#2 (in the first stage, HDPE or CP#2 was degraded in a 150-cm<sup>3</sup> autoclave at 435°C, 1 hour, 800 rpm, under N<sub>2</sub> or H<sub>2</sub>; in the second stage, DECS-6 coal was liquefied with Plastic-Derived-Liquids obtained from the 1st stage as solvents in a 27-cm<sup>3</sup> tubing reactor at 400°C, ~2000 psig H<sub>2</sub>, 60 minutes, 160 rpm (solvent: dry coal = 2:1, weight ratio))

Solvent	Gasoline (<200°C)	Kerosene (200-275°C)	Gas Oil (275-325°C)	Heavy Gas Oil (325-400°C)	Vacuum Gas Oil (400-538°C)	Vacuum Residue (>538°C)
None	8.2	24.0	15.9	19.7	30.7	1.5
Waste Oil	3.0	5.3	5.7	22.7	47.0	16.3
A-1 Oil	26.9	27.5	15.9	16.6	13.1	0.0
A-2 Oil	42.6	28.0	12.3	11.4	5.7	0.0
A-4 Oil	31.3	26.0	14.7	16.1	11.9	0.0
A-5 Oil	35.0	27.1	14.1	14.1	9.7	0.0
A-7 Oil	22.3	22.6	14.2	17.5	16.9	6.5
A-8 Oil	59.6	24.3	7.9	5.6	2.6	0.0
A-10 Oil	26.5	22.3	13.8	17.8	18.0	1.6
A-11 Oil	38.0	25.2	12.8	13.7	10.3	0.0

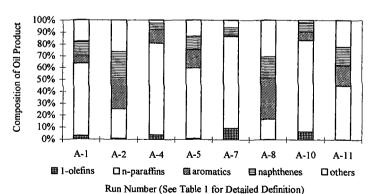


Figure 1. Effects of Catalyst (HZSM-5) and Reaction Atmosphere (N<sub>2</sub> or H<sub>2</sub>) on Composition of Oil Products Obtained from Degradation of HDPE or CP#2 in a 150-cm<sup>3</sup> Autoclave at 435°C, 800 rpm, for a Reaction Time of 60 Minutes